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Comparisons of some NIST fixed-point cells with similar cells of other standards laboratories

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Abstract. In this paper we present results of international comparisons of fixed-point cells of some of the defining fixed-point materials of the International Temperature Scale of 1990. These comparisons involved cells from seven national laboratories, although in some cases only one type of fixed-point material was compared. Except for silver cells, the agreement among cells of the same defining fixed-point material from the various laboratories was to within 1 mK. The expanded uncertainties ($k=2$) of the comparison measurements were 12 μK for Ga, 18 μK for H_2O, 27 μK for Sn, 35 μK for In, 37 μK for Zn, 42 μK for Al, and 55 μK for Ag.

1. Introduction

Over the past few years, the thermometry groups of the Centro Nacional de Metrología (CENAM); the Center for Measurement Standards of the Industrial Technology Research Institute (CMS/ITRI); the Istituto di Metrologia "G. Colonnetti" (IMGC); the National Institute of Metrology (NIM); the National Metrology Institute (UME); and the Physikalisch-Technische Bundesanstalt (PTB) have participated in bilateral comparisons of fixed-point cells with the Thermometry Group of the National Institute of Standards and Technology (NIST). Some or all of the following fixed points have been compared: triple point of water (TPW), triple/melting point of Ga, and freezing points of In, Sn, Zn, Al and Ag. Those cells contained high-purity materials, making their respective fixed-point temperatures suitable for use as defining fixed points of the International Temperature Scale of 1990 (ITS-90) [1]. All of the comparison measurements were conducted in the laboratories of the Thermometry Group of NIST. Through these bilateral comparisons with NIST, the other laboratories obtained an indirect comparison of their fixed-point cells with one another. In most cases, only one or two fixed-point cells from any one laboratory were compared. Only in the case of CENAM were fixed-point cells for all seven defining fixed points from 273,16 K to 1234,93 K compared, i.e. TPW cells, Ga melting-point cells (in fact, Ga cells operated in the triple-point mode were used (see Section 3)), and In, Sn, Zn, Al and Ag freezing-point cells. In the case of IMGC, Sn, Zn, Al and Ag freezing-point cells were compared, the results of some comparisons already being published elsewhere [2]. One TPW cell and one Sn freezing-point cell were compared with UME. Only Ag freezing-point cells were compared with PTB and CMS/ITRI. The results of the comparison with PTB have already been reported [3], but are included here for completeness. Only TPW cells were compared with NIM. The results of all these comparisons are presented here, as are the techniques employed for the measurements.

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2. Experimental details

In this section, we present details of the apparatus in which the cells were compared, the measuring instrumentation, and the fixed-point cells and their assemblies. Details of the comparisons are presented in Section 3.

2.1 Triple point of water maintenance bath

The TPW cells were kept in a water bath of commercial manufacture that was controlled at approximately 0,007 °C. With this bath, the mantles of the TPW cells could be maintained for months.

2.2 Furnaces

The two furnaces used for comparison of pairs of metal fixed-point cells were, for all practical purposes, identical. The temperature gradients in all the NIST furnaces used to compare the fixed-point cells were small over the volume of the fixed-point samples: the range of temperature values along the axis of a fixed-point sample in its respective furnace was < 10 mK over the length of the ingot, as measured in a cell held at a temperature a few kelvins below the phase-transition temperature. This temperature uniformity ensured correct formation and advancement of the solid/liquid interfaces in the samples. The measurements at NIST were made using (high-temperature) standard platinum resistance thermometers ((HT)SPRTs) for temperatures in the range 25 °C to 960 °C. Also, the variations in the furnace temperatures (< 10 mK) were small with respect to the difference between the temperature of the furnace and the fixed-point temperature of the sample being maintained at the transition from the liquid (solid) to the solid (liquid) state. With the exception of the furnace for Ga triple/melting-point cells, all NIST furnaces were of three-zone type (for In, Sn and Zn) or were fitted with sodium heat-pipe liners (for Al and Ag) [4-7]. All furnaces were dc powered to eliminate ac pick-up problems. Each zone of the three-zone furnaces was controlled automatically, with the end zones controlled relative to the main zone.

The two Ga furnaces, which have aluminium containers (thick-wall aluminium tubes) filled with a light mineral oil for good thermal contact, were maintained at a temperature of 29,93 °C. With these furnaces, the Ga phase transition could be maintained for at least four months. The Ga phase-transition-preparation furnace, similarly constructed, was maintained at 40 °C.

An (HT)SPRT pre-heat (annealing) furnace was associated with each main furnace. Each of the auxiliary (or annealing) furnaces associated with the Al and Ag fixed-point furnaces contained a closed-end protection tube of Pt in which the thermometer was placed [7]. These Pt tubes were used to minimize contamination of the (HT)SPRTs by metallic impurities that might be present in the furnace during the heat treatment of the thermometers [8], i.e. while heating them to, annealing them at, and cooling them from the Al and the Ag freezing-point temperatures. Protection of the (HT)SPRTs in this way also helped to minimize contamination of the fixed-point cells.

2.3 Platinum resistance thermometers

For comparisons at the TPW, Ga triple point, and the In, Sn, Zn and Al freezing points, NIST long-stem 25,5 Ω check SPRTs associated with each fixed-point temperature were used. For the Al comparison, that thermometer was a 25,5 Ω Chino SPRT, with fused-silica supports for the Pt coil and leads. For the comparison of Ag cells, except for the one from IMGC, the NIST check HTSPRT, a D.I. Mendeleyev Institute of Metrology (VNIIM)-manufactured 0,59 Ω HTSPRT [9], was used. For the comparison of the IMGC and NIST Ag cells, a 0,38 Ω NBS-constructed HTSPRT, a 0,24 Ω NIM-made HTSPRT and a 2,5 Ω Chino HTSPRT were used.

By using the check (HT)SPRT associated with a given fixed point, a check on the cells was obtained at the beginning of each freeze (or melt) and throughout the comparison measurements. This gave assurances that the freeze (or melt) had progressed as expected.

A list of the (HT)SPRTs used in the comparisons is given in Table 1.

Table 1. NIST (HT)SPRTs used in the comparisons of fixed-point cells.

Fixed-point cell tested	Manufacturer/ model (HT)SPRT	Serial number
TPW	Leeds & Northrup 8167	1881990
Ga	Leeds & Northrup 8163	1803100
In	Chino R800-2	RS59A-7
Sn	Leeds & Northrup 8167	1868894
Zn	Chino R800-2	RS7YA-5
Al	Chino R800-2	RS87A-5
Ag	VNIIM	BTC-MN0002
Ag (IMGC)	Chino R800-3	RS48A-5 (2,5 Ω)
	NIM	80179 (0,24 Ω)
	NBS	8205 (0,38 Ω)

2.4 Resistance-ratio bridge

The first comparison measurements conducted were those of the IMGC and NIST Ag cells. They were performed using a 400 Hz Cutkosky resistance-ratio bridge [10], an automatic-balancing 30 Hz Cutkosky resistance-ratio bridge [11], and a Guildline 9975 current comparator (a dc bridge). The reference resistors for these bridges were maintained at a constant temperature, (39,00±0,01) °C for the 30 Hz Cutkosky bridge and (29,00±0,01) °C for both the 400 Hz Cutkosky bridge and the Guildline bridge. Measurements of the 0,24 Ω and 0,38 Ω HTSPRTs with the 30 Hz Cutkosky bridge were conducted at 4 mA and 8 mA of excitation current; at 2 mA and $2\sqrt{2}$ mA with the 400 Hz Cutkosky bridge; and at 10 mA, $10\sqrt{2}$ mA, and 30 mA with the Guildline bridge. For the 2,5 Ω HTSPRT, the excitation currents were 2 mA and 4 mA with the 30 Hz Cutkosky bridge; 2 mA and $2\sqrt{2}$ mA with the 400 Hz Cutkosky bridge; and 1 mA, $\sqrt{2}$ mA and 3 mA with the Guildline bridge. The two or three

Table 2. List of fixed-point cells, and their characteristics, used in the comparisons. Also, the depth of immersion over which the ITS-90 designated hydrostatic-head effect was observed is listed in column 6.

Laboratory	Fixed point		Fixed-point cell serial no.	Purity[a]/%	Cell immersion depth[b]/cm	Measured hydrostatic head[b]/cm
NIST	H_2O	TP	A-13-1289		26,5	8
CENAM	H_2O	TP	CENAM 2		27	8
NIM	H_2O	TP	237		26	7
UME	H_2O	TP	UME4		22,5	4
NIST	Ga	TP	Ga-1	99,999 99+	13	5
CENAM	Ga	TP	Ga 94-1	99,999 99_5	18	10
NIST	In	FP	In-1	99,999 9+	19	9
CENAM	In	FP	In 93-1	99,999 95	18	8
NIST	Sn	FP	Sn 75A	99,999 9	18	10
NIST	Sn	FP	Sn 88A	99,999 99_8	18	10
CENAM	Sn	FP	Sn 93-3	99,999 92_5	18	10
IMGC	Sn	FP	Sn 88B	99,999 99_8	18	10
UME	Sn	FP	079	99,999 9	17,5	4
NIST	Zn	FP	Zn 86D	99,999 9	18	8
NIST	Zn	FP	Zn 89C	99,999 95_3	18	8
NIST	Zn	FP	Zn 93-4	99,999 96_8	18	8
CENAM	Zn	FP	Zn 93-3	99,999 97	18	8
IMGC	Zn	FP	Zn 89B	99,999 95_3	18	8
NIST	Al	FP	A-2	99,999 8	16,7	5
NIST	Al	FP	Al 78-1	99,999 9	16,7	5
NIST	Al	FP	Al 94-2	99,999 96	18	6
CENAM	Al	FP	*Al 93-4*	*99,999 90*	*18*	*6*
IMGC	Al	FP	Al Col	99,999 9	15,5	4
NIST	Ag	FP	Ag 79-1	99,999 9	16,7	3
NIST	Ag	FP	Ag 90-3	99,999 93_1	18	4
NIST	Ag	FP	Ag 92-1	99,999 97_4	18	4
NIST	Ag	FP	Ag 92-4	99,999 97_4	18	4
CENAM	Ag	FP	Ag 94-1	99,999 96_9	18	4
CMS/ITRI	Ag	FP	CMS Ag 92-1	99,999 9	14,8	2
IMGC	Ag	FP	Ag Lei3	99,999 9	15,4[c]	—[d]
PTB	Ag	FP	Ag 6	99,999 9	16,5	—[d]

a. Values taken from assay reports from suppliers of the metals.
b. Relative to the middle of the (HT)SPRT platinum sensor.
c. Based on the average of the HTSPRTs used in the comparison.
d. Immersion study not performed during the time of the comparison experiments.

excitation currents were used in order to permit analysis of the results at zero-power dissipation in the HTSPRTs.

The electronic measurement equipment for the subsequent comparisons included an ASL F-18 resistance-ratio bridge, operating at a frequency of 30 Hz, and temperature-controlled Tinsley 5685A 10 Ω and 100 Ω reference resistors. These reference resistors were maintained at a temperature of (25,00±0,010) °C. Measurements of the thermometer resistance were conducted at two excitation currents, 1 mA and $\sqrt{2}$ mA with 25,5 Ω SPRTs and $5\sqrt{2}$ mA and 10 mA with the 0,59 Ω HTSPRT, to allow analysis of the results at zero-power dissipation. A computer-controlled data acquisition system was used to acquire the ASL F-18 bridge readings through the use of an *IEEE-488* bus.

2.5 *Fixed-point cells and their assemblies*

The cells (and their characteristics) of the various laboratories that were compared are listed in Table 2.

The TPW cells belonging to NIST were obtained commercially from Jarrett Instrument Co. The TPW cells belonging to CENAM [12], NIM and UME were constructed in their respective laboratories.

Except for the TPW cells, the other NIST fixed-point cells had been constructed in-house from the various components. The design of the metal fixed-point cells and their assemblies has been described previously [3-7, 13, 14]. Since the fixed-point cell assembly for Ag and Al cells is extremely important if adequate immersion is to be obtained, and especially so for Ag, the cell assembly used in those comparisons is described again.

None of the NIST metal fixed-point cells was of the sealed-cell type. The NIST Ga cell consisted of high-purity Ga in an all-plastic container [15, 16]. Both the NIST and the CENAM Ga cells were semi-open and each could be used as either a melting-point or a triple-point cell but they were used in the triple-point mode.

The NIST In cell has been described elsewhere [17]. The valve of the NIST In-1 cell was closed with a pressure of 101,3 kPa of He in the cell at the freezing-point temperature. The CENAM cell was constructed using the same design as that of the NIST Sn and Zn cells and was controlled at a pressure of 101,3 kPa.

For Sn and Zn, the crucibles containing the high-purity metals were in fixed-point-cell assemblies [4-7, 13], within which the atmosphere was He. The pressure of the He was controlled at approximately 101 325 Pa (one standard atmosphere) and corrections were made for differences from 101 325 Pa. The NIST cell Sn 88A and the IMGC Sn cell contained tin of $99{,}999\,99_8$ % purity, as analysed by the supplier.

The NIST cell Zn 89C and the IMGC cell Zn 89B were two of three fixed-point cells constructed from SRM 740a. This SRM zinc was specially prepared for NIST (by Johnson Matthey, Inc., Spokane, WA, USA), was all of one lot and was analysed by the supplier to be $99{,}999\,95_3$ % pure. The freezing-point temperatures of all three of the cells containing the SRM zinc agreed to within ±0,15 mK, as determined by direct comparison.

The NIST Al and Ag fixed-point cells consisted of the high-purity metals in high-purity-graphite crucibles, which were enclosed in fused-silica glass containers that were connected by a matte-finished, small-diameter, fused-silica tube to a valve outside the fixed-point cell assembly [3, 6, 7]. Purified and dried Ar gas at approximately 101,3 kPa filled the empty space inside the fused-silica enclosure. Each of the enclosing fused-silica glass containers for the crucibles had a re-entrant fused-silica glass well with a matte finish.

The NIST cell Al 94-2 was one of three NIST fixed-point cells constructed from SRM 1744. This SRM aluminium [14] was specially prepared for NIST (by Johnson Matthey, Inc., Spokane, WA, USA), was all of one lot and was analysed by the supplier to be 99,999 96 % pure. The freezing-point temperatures of all three of the NIST cells containing the SRM aluminium agreed to within ±0,1 mK, as determined by direct comparison.

The NIST cells Ag 92-1 and Ag 92-4 were two of three NIST fixed-point cells constructed from SRM 1746. This SRM silver also was specially prepared for NIST (by Johnson Matthey, Inc., Spokane, WA, USA), was all of one lot and was analysed by the supplier to be $99{,}999\,97_4$ % pure. The freezing-point temperatures of all three of the NIST cells containing the SRM silver agreed to within ±0,1 mK, as determined by direct comparison.

The NIST Al and Ag fixed-point cells were semi-open cells [3, 6, 7], but before measurements on them were made, they had been sealed at the freezing-point temperature with 101,3 kPa of purified Ar. The PTB Ag cell had been sealed previously at a pressure of 95,0 kPa of Ar.

The immersion depths for the cells are shown in Table 2.

The NIST Al and Ag freezing-point cell *assemblies*, which are placed in sodium heat-pipe furnaces for realization and comparison measurements, were used for all Al and Ag cell comparisons [3, 6, 7]. This cell assembly consisted of the Ag or Al cell in a 61 cm long closed-end Inconel tube that contained a small cushion (about 5 mm thick) of Fiberfrax blanket at the bottom. From the top of the re-entrant well of the cell to 1 cm above the top of the Inconel tube, there was a matte-finished, silica-glass guide tube for the thermometer. Immediately above the top of the cell, there was a 1 cm air space followed by thirteen Inconel radiation shields, each 0,08 mm thick, held 1 cm apart by silica-glass tube spacers and with only air in the remaining space between them. The remaining top 18 cm of space in the Inconel tube was filled with disks of Fiberfrax blanket.

The Ag cells of CMS/ITRI, IMGC and PTB, and the Al cell of IMGC, were of the sealed-cell design and were constructed in their respective laboratories. Those sealed cells of Ag and Al were mounted at NIST in the NIST cell assembly for comparison. By this procedure, a good comparison of the samples was obtained. In a sense, however, that procedure was unfortunate since a better comparison of the fixed-point realizations at the relevant institutions could have been made if the various laboratories had provided their cells in their own cell assemblies [3], even though the furnace characteristics of the different laboratories might be substantially different.

Except for the Ga fixed-point cell, the fixed-point cells of CENAM were of the same design as those of NIST. The CENAM Ga cells (designed for use in the triple-point mode) merged the NIST and the National Research Laboratory of Metrology (Japan) designs [16, 18]. All of the CENAM metal cells were constructed at NIST.

The Sn and Zn fixed-point cells of IMGC, Sn 88B and Zn 89B, respectively, were of the same design as those of NIST and were constructed at NIST. After those cells were compared with NIST cells at NIST, they were transferred by hand to IMGC. Then, after comparisons (by IMGC) at IMGC of those cells with other IMGC Sn and Zn cells, respectively, those NIST-constructed cells became the IMGC Sn and Zn reference cells.

The Sn fixed-point cell of UME, which had been purchased from Engelhard, was of a sealed-cell design. The glass enclosure had broken at UME, with no apparent damage to the crucible or to the ingot of Sn. Subsequently, the cell was brought to NIST where the crucible containing the Sn ingot was placed in the usual NIST assembly [4-7] for comparison measurements.

3. Description of the comparisons

Over the years, NIST has acquired and/or constructed numerous fixed-point cells for its use. When new TPW cells are acquired and/or when new metal cells are constructed, the cells are compared with the other cells in stock. Also, as part of an internal measurement assurance programme, those cells used as laboratory standards are periodically compared to ensure that they have not changed with time. These comparisons are performed in the same way as described below for the comparison of the NIST cells with the cells of the other national laboratories. NIST maintains records of these comparisons of its TPW cells (of which, at the present time, there are nineteen) and of its other fixed-point cells [19] and the results of these comparisons relevant to the work presented here are given in Table 3.

It has been reported [20] that the method of preparation of ice mantles of TPW cells is immaterial, the final temperature of the mantles being independent of the technique of their preparation. For the comparisons of TPW cells reported here, the ice mantles of the NIST cells were prepared in advance using immersion coolers. The mantles extended the full length of the water columns of the cells. After completion of the preparation of the ice mantles, the alcohol was removed from the wells of the cells, the cells were stoppered, and they were then placed in a maintenance bath, which was maintained at about 0,007 °C. About 30 min after the completion of the preparation of a mantle, the re-entrant well of the cell was filled with maintenance-bath water. Comparison measurements of the cell containing this new mantle with another NIST TPW cell containing a mantle several weeks old began about 30 min after the well had been filled with the cold bath water. Measurements were made on the two cells every day, Monday through Friday, for the next two weeks to confirm the temperature stability of the new mantle after 1 to 3 days.

The mantle of the NIST TPW cell used in a comparison with a cell from another laboratory had been prepared at least four weeks before the comparison measurements began. The NIM cell was compared twice with the NIST reference TPW cell. For the first comparison, the ice mantle of the NIM cell was prepared using a liquid-nitrogen immersion cooler; for the second comparison, the ice mantle was prepared using a solid-CO_2 immersion cooler. Both the CENAM and UME TPW cells were compared once with the NIST reference cell and their mantles were prepared using a solid-CO_2 immersion cooler.

Prior to the beginning of the comparison measurements, an *inner* liquid/solid interface was prepared in the cells by inserting an Al rod into the well (a glass rod would have worked just as well). This rod was initially at room temperature, and was left in the well until the mantle could rotate freely. The mantles were checked after each comparison measurement to see that they were still free to rotate.

When the space between the SPRT and the re-entrant well of the TPW cell being compared with the NIST cell permitted, an Al bushing (approximately 5 cm long, with an inner taper to guide the SPRT into the bushing) was inserted into the cell to improve the thermal contact between the SPRT and the *inner* liquid/solid interface of the ice mantle. The bushings were cooled to approximately 0 °C before being placed in the wells. A small foam pad was placed in the bottom of each well prior to insertion of the bushing. This pad prevented mechanical shock to a SPRT when

Table 3. Observed temperature differences expressed as ΔT (Lab, NIST) = [T(Lab) − T(NIST)].

			Cell serial number		
Laboratory	Fixed point		Laboratory	NIST[a]	ΔT/mK
NIST	Sn	FP	Sn 88A	Sn 75A	0,71
	Zn	FP	Zn 89C	Zn 86D	0,04
	Zn	FP	Zn 93-4	Zn 89C	0,04
	Al	FP	Al 78-1	A-2	1,22
	Al	FP	Al 94-2	Al 78-1	0,32
	Ag	FP	Ag 90-3	Ag 79-1	2,50
	Ag	FP	Ag 92-4	Ag 90-3	0,91
	Ag	FP	Ag 92-1	Ag 92-4	0,10
CENAM	H_2O	TP	CENAM 2	A-13-1289	0,00[b]
	Ga	TP	Ga 94-1	Ga-1	0,03
	In	FP	In 93-1	In-1	− 0,02
	Sn	FP	Sn 93-3	Sn 88A	0,06
	Zn	FP	Zn 93-3	Zn 93-4 (Zn 89C)[c]	− 0,35
	Al	FP	Al 93-4	Al 94-2 (Al 78-1)[c]	− 0,76
	Ag	FP	Ag 94-1	Ag 92-1 (Ag 92-4)[c]	− 0,25
CMS/ITRI	Ag	FP	CMS Ag 92-1	Ag 92-1	− 2,99
IMGC	Sn	FP	Sn 88B	Sn 88A (Sn 75A)[c]	− 0,01
	Zn	FP	Zn 89B	Zn 93-4 (Zn 86D)[c]	− 0,18
	Al	FP	Al Col[d]	Al 94-2 (A-2)[c]	− 0,80
	Ag	FP	Ag Lei3[d]	Ag 92-1 (Ag 79-1)[c]	− 8,81
NIM	H_2O	TP	237	A-13-1289	0,00[b]
PTB	Ag	FP	Ag 6	Ag 92-1	− 0,22
UME	H_2O	TP	UME4	A-13-1289	− 0,04[b]
	Sn	FP	079	Sn 88A	− 0,57

a. Zn 89B and Zn 89C are from SRM 740a; A-2 is from SRM 44f; Al 94-2 is from SRM 1744; Ag 92-1 and Ag 92-4 are from SRM 1746.

b. Average of last five differences between test cell and NIST cell.

c. The cell identified in parentheses refers to the NIST cell actually used in the comparison measurements, i.e. the transfer cell. The other cell identified in this row is the NIST reference cell. The value of ΔT in this row was derived by using the NIST reference cell.

d. IMGC transfer cell.

it was inserted into the well. The pad and bushing were removed at the end of the day and re-inserted prior to the next set of measurements on the cell. The use of an Al bushing and a small foam pad in a TPW cell is standard operating procedure at NIST.

The usual precautions were observed to prevent light piping of radiation into the TPW cells [21].

The SPRT used in the comparison was cooled in ice water prior to its insertion into the first TPW cell. After placement of the SPRT in the cell, at least 30 min were allowed to elapse before measurements began in order to ensure that the SPRT attained equilibrium with the *inner* liquid/solid interface of the ice mantle of the cell.

Measurements of the SPRT resistance were made at two measuring currents so that the resistance value could be calculated for zero power dissipation. Following the measurement at the second thermometer current, measurement at the first current was repeated at least once for each cell to check for thermal equilibrium and also for repeatability. This procedure was also followed for the comparison of the metal cells. For the measurements on TPW cells (for which 25,5 Ω SPRTs were used), the repeat measurement always agreed with the first measurement to within 0,02 mK.

The SPRT resistance value measured in a test cell was compared with that obtained in the NIST reference cell, and expressed as a ratio of the zero-power resistance values. As long as the NIST reference cell is stable, this procedure should eliminate the effects of slow drifts and other changes in the measurement equipment (e.g. drifts in the reference resistor, recalibration of the reference resistor, drift in the SPRT, accidental bumping of the SPRT at times other than during the experiment on a given day, and even the substitution of other SPRTs, making the daily experiment independent of the reference resistor and SPRT used). The order of measurement of the TPW cells was the NIST TPW cell, the test cell and then a repeat measurement of the NIST cell. The difference between the test TPW cell and the NIST TPW cell, if any, was obtained by averaging the differences between the cells for the last five days of measurements. Figure 1 displays a typical example of measurement data as a function of time.

NIST maintains records not only of comparisons, but also of melting and freezing curves, of its own metal fixed-point cells (see Section 3). NIST has at least two nearly-identical furnaces for the realization of each of the defining fixed points. Prior to a comparison of a NIST metal fixed-point cell with a similar cell from another laboratory, melting and freezing curves were obtained for at least one of each type of cell from the other laboratory in order to determine the melting and freezing behaviour of that laboratory's cell in the NIST furnace used for that cell. From the characteristics of the freezing curves of those fixed points realized by freezing, the length of time required for attainment of equilibrium after recalescence and subsequent formation of the inner mantle on the thermometer well was

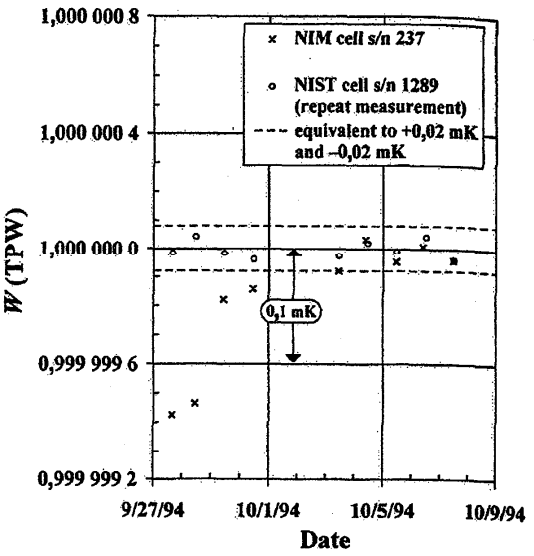

Figure 1. Results of the comparison of the NIM and the NIST TPW cells over a two-week period. The temperature difference between the NIM cell and the NIST cell given in Table 3 is the average of the differences obtained over the last five days of measurements. The mantle of the NIM cell was prepared using a liquid-nitrogen type immersion cooler whereas the mantle of the NIST cell was prepared using a solid CO_2 type immersion cooler. The initial temperature of the cells before mantle preparation was 274 K.

determined so that equilibration times could be taken into account in the comparison experiments.

Except for Ga triple-point cells, all comparisons of metal fixed-point cells were made during freezing of the samples. The difference between the freezing-point temperature of the sample and the temperature of the furnace during the freeze was 0,5 K to 0,75 K, small enough for the plateau of a freezing curve to have a minimum duration of 16 h to 24 h. In the case of Ag, however, the duration was somewhat shorter, being only 10 h to 12 h. For Ga, comparisons were made during melting of the samples.

With the exception of the IMGC and the PTB Ag freezing-point cells, the immersion characteristics of the long-stem (HT)SPRTs used in the comparisons of the cells were determined in each type of fixed-point cell that was compared, with the cells located in the NIST fixed-point cell assembly and furnace used for the comparison. The measurements (obtained upon *insertion* of the (HT)SPRT into the cell) showed agreement with the ITS-90 designated hydrostatic-head effect over at least the bottom-most 3 cm of immersion in the cell. See Figure 2 for a typical example of an immersion curve showing the hydrostatic-head effect. The depth of immersion over which the ITS-90 designated hydrostatic-head effect was observed in the various cells is listed in Table 2.

Since the immersion depth of the PTB Ag freezing-point cell was 1,5 cm shorter than that of the NIST

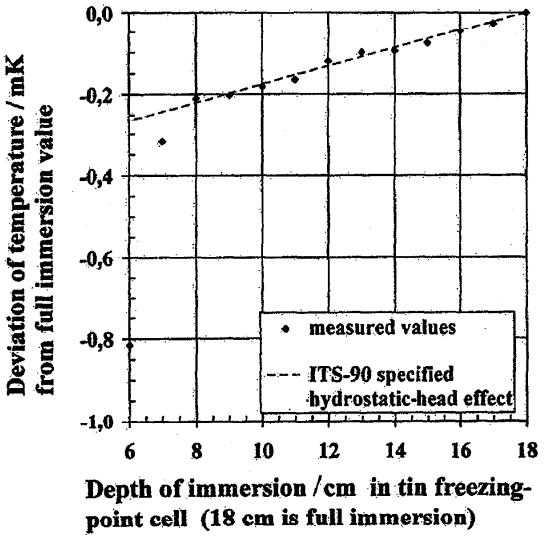

Figure 2. Immersion curve showing the effect of the hydrostatic head of the liquid Sn in the CENAM Sn cell on the SPRT reading upon the insertion of the SPRT into the cell. The cell was located in a NIST three-zone furnace. The ITS-90 specified hydrostatic-head effect is shown as a dashed line.

cells, the HTSPRT immersion in the PTB cell in the NIST assembly should have been able to exhibit the hydrostatic-head effect over the bottom-most 2,5 cm, although an immersion study of it was not performed during the comparison experiments. Similarly, the IMGC cell had an immersion depth of 15,4 cm, i.e. 2,6 cm shorter than that of the NIST cells, so the HTSPRT immersion in that cell in the NIST assembly should have been able to exhibit the hydrostatic-head effect over the bottom-most 1,4 cm, although no immersion study was performed during the comparison experiments.

The procedure for a comparison of Ga cells was as follows. The two liquid/solid interfaces of a Ga sample were prepared by placing the cell, at room temperature, in the Ga fixed-point preparation furnace at 40 °C, and leaving the cell there for 30 min. This produced the *outer* interface. Throughout that time, an immersion heater placed in the well of the cell supplied sufficient heat to keep the oil in the well at 40 °C. That produced the *inner* interface. After 30 minutes, the cell was transferred to one of the Ga fixed-point furnaces (maintained at 29,93 °C). The second cell was treated similarly. Then, at least another hour was allowed to elapse before comparison measurements were begun.

The procedure for a comparison of the other metal (freezing-point) cells was as follows. The two cells to be compared were placed in their respective (essentially identical) furnaces, with the cells and the furnaces at room temperature. The furnaces containing the cells were then heated fairly slowly to a temperature approximately 5 K higher than the melting-point temperature of the metal samples. This required about 3 h for the In furnaces, 6 h for the Sn furnaces, 10 h for the Zn furnaces, 24 h for the Al furnaces and 48 h for the Ag furnaces. Then, the two cells to be compared were kept in their respective furnaces overnight at temperatures approximately 5 K higher than the melting-point temperature of the metal samples. This ensured that the metal samples were not only completely melted but also had a uniform distribution of any impurities in the liquid phases.

In most cases, at the time of the actual comparisons, the NIST cell to be compared was already being maintained at a temperature a few kelvins below the freezing-point temperature. Consequently, in its preparation for comparison, the cell was heated to about 5 K above the melting-point temperature and kept at that temperature overnight.

For comparison of the fixed points, the usual NIST procedure [4-7] of inducing two liquid/solid interfaces in a sample was followed. The freezing experiments were started with the completely molten samples that had been kept overnight at approximately 5 K above their freezing-point temperature. The check (HT)SPRT for the sample of interest was removed from the pre-heat furnace and it was inserted into the well of one of the two cells to be compared and permitted to come into equilibrium with the sample. Typically, 30 min were allowed for this equilibration. Then, a layer of solid (for the *outer* liquid/solid interface) was formed at the crucible wall of the cell by setting the furnace temperature to a value about 5 K below the freezing-point temperature and permitting the sample to supercool until recalescence was observed with the (HT)SPRT. Then, the furnace temperature was set to 0,5 K to 0,75 K below the freezing-point temperature and the (HT)SPRT was removed and inserted into the second cell. Then, an *inner* mantle of metal was induced on the thermometer well of the first cell by inserting successively two cool (room temperature) silica-glass rods. For In, the two rods were inserted successively in the well for about 3 min each; for Zn, about 5 min each. For Al and Ag, one rod was used to prepare the inner mantle; a rod, initially at room temperature, was inserted in the well for one minute, removed to cool for one minute, inserted again for one minute and then removed. The two interfaces of the second cell were induced in the same way.

In the case of tin, the freeze for each cell was nucleated by rapid cooling outside the furnace. After the tin had melted overnight in a furnace maintained at approximately 5 K above the melting point of the sample, the check SPRT was inserted into the cell well and when the SPRT indicated that the temperature of the molten sample was about 5 K above the freezing-point temperature, the furnace was set to control at 0,5 K to 0,75 K below the freezing-point temperature. The sample cooled and when the SPRT indicated that the cell temperature was within about 10 mK of the freezing-

point value, the cell and the SPRT were withdrawn from the furnace. The cell cooled rapidly and when the SPRT detected recalescence, the cell was replaced in the furnace. In order to freeze a thin mantle of solid around the thermometer well, the SPRT was withdrawn from the cell well, and two fused-silica glass rods, each initially at room temperature, were inserted successively in the well for about 3 min each.

The induced freezes, or melts, of the two cells being compared were established in such a way that the time from nucleation of the freeze (or the beginning of the melt) to the first measurement on the first cell was the same as the time from nucleation of the freeze (or the beginning of the melt) to the first measurement on the second cell. Thus, comparison measurements on the two cells, each in its own furnace, were made at approximately the same liquid/solid ratio of the metal samples. Measurements were made alternately in the two cells. For the Ga fixed point, the temperature of the plateau region of the melting curves was of such constancy in time that the temperature values of the plateaux were for all practical purposes independent of the ratio of liquid to solid.

For the comparison of the cells, the (HT)SPRT was quickly, but gently, transferred directly back and forth between the two cells, each transfer following measurements in a cell at two excitation currents. Following the measurement at the second thermometer current, measurement at the first current was repeated at least once for each cell to check for thermal equilibrium and, also, for repeatability. For these measurements with 25,5 Ω SPRTs, the repeat measurement always agreed with the first measurement to within the equivalent of 0,02 mK (see Section 2.4). For the 0,6 Ω HTSPRT at the Ag freezing point, the repeat measurement agreed with the first measurement to within the equivalent of 0,04 mK (see Section 2.4).

Three pairs of measurements, with the (HT)SPRT being transferred directly from one cell to the other, were made on each of three separate freezes (or melts, in the case of Ga) of the two fixed-point cells being compared. (For the comparison of the UME Sn cell with the NIST Sn cell, only one freeze was obtained, and for the comparison of the CMS/ITRI Ag cell with the NIST Ag cell, only two freezes were obtained.) Although only the first of the three pairs of measurements on a given freeze of a sample was used for the comparison, the other two measurements on the cell provided information on the progress of the freezes (see Figure 3). (Ideally, the difference between the measurements of each of the pairs would be identical.) Only the first 25 % of the freezing curves was used for the comparisons. To remove any bias in the measurements, the cell measured first in the sequence was changed from comparison to comparison.

After measurements at the In, Sn or Zn fixed points were completed, the SPRT was removed directly from the fixed-point cell into the room-temperature environment and measured at the TPW as soon as possible to minimize the effects of changes in the oxidation state of the thermometer.

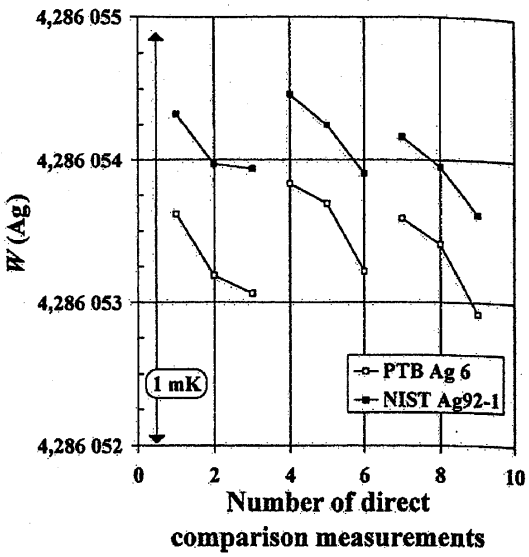

Figure 3. Results of the direct comparison of the PTB Ag cell, Ag 6, with the NIST Ag cell, Ag 92-1. Three pairs of measurements on three separate freezes are shown.

In preparation for Ag cell comparisons, the HTSPRT to be used was placed in an auxiliary furnace at approximately 500 °C and then the temperature was increased from 500 °C to about 975 °C over a period of about 45 min to 60 min. Similarly, the SPRT to be used at the Al fixed-point temperature was heated to about 675 °C in a comparable length of time.

The (HT)SPRT was maintained at either 975 °C (for Ag) or 675 °C (for Al) for about 30 min before it was quickly, but gently, transferred to the first Ag or Al fixed-point cell being compared.

After measurements were completed on the two Al fixed-point cells, the SPRT was quickly, but gently, transferred to an annealing furnace at a temperature of about 675 °C, maintained at that temperature for at least 30 min, and then cooled over a 3,5 h to 4 h period from 675 °C to 500 °C, at which temperature the SPRT was removed directly from the furnace into the room-temperature environment and measured at the TPW as soon as possible.

After measurements were completed on the two Ag fixed-point cells, the HTSPRT was quickly, but gently, transferred to the annealing furnace at a temperature of about 975 °C, maintained at that temperature for at least 30 min, and then cooled over a 4 h or longer period from 975 °C to 500 °C, at which temperature the HTSPRT was removed directly from the furnace into the room-temperature environment and measured at the TPW as soon as possible.

Prior to the comparison with the PTB silver cell, NIST cell Ag 92-1 was directly compared with NIST cell Ag 92-4. After comparison measurements with the

PTB silver cell, the two NIST cells were compared again to ascertain if NIST cell Ag 92-1 had changed during the course of the measurements on the PTB cell. Measurements made prior to the PTB comparison indicated that the freezing-point temperature of NIST cell Ag 92-1 was $0{,}1_0$ mK higher than that of NIST cell Ag 92-4. The results from the direct comparison of NIST cells Ag 92-1 and Ag 92-4 after completion of the comparison with the PTB cell showed that the freezing-point temperature of cell Ag 92-1 was $0{,}1_1$ mK higher than that of cell Ag 92-4, i.e. unchanged to within the measurement uncertainty. This confirmed that the freezing-point temperature of NIST cell Ag 92-1 did not change during the course of the PTB-NIST fixed-point cell comparison measurements.

4. Uncertainty of measurements

The expanded uncertainty U assigned to the measurements was calculated from the equation

$$U = k\sqrt{s^2 + \sum u(i)^2}, \qquad (1)$$

where k is the coverage factor, s is the type A standard uncertainty based on the calculated standard deviation of the mean of the measurements and $u(i)$ is the estimated type B standard uncertainty for each known component in the measurement process that cannot be directly measured [22].

Since the experiments reported here are direct comparisons of fixed-point cells, all systematic effects are incorporated in the type A standard uncertainties. The type A standard uncertainty for these experiments was estimated to be the standard deviation of the mean of the temperature differences of the two cells being compared, as determined from the W values obtained from the (HT)SPRT readings in each of the two cells. The value of the type A standard uncertainty for the NIST measurements was at most $0{,}02_3$ mK, except for one case in which there was an obvious "bump" of the thermometer for one measurement in one cell and the value was $0{,}02_8$ mK. There were two known contributions to the type A standard uncertainty; one from the instrumental measurements themselves and the second from handling of the (HT)SPRT (transferring the thermometer from cell to cell). The contribution from instrumentation was equivalent to at most $0{,}00_9$ mK. The remainder, and the largest contribution, to the type A standard uncertainty came from handling the (HT)SPRT, with a maximum contribution of $0{,}02_1$ mK, except for the measurements during which the SPRT was bumped and the value was $0{,}02_8$ mK.

Apart from the systematic effects that, because of the nature of the experiments, were incorporated in the type A standard uncertainties, there were three known sources of type B standard uncertainties. These were the uncertainty in the exact immersion depth of the (HT)SPRT due to the uncertainty in the position of the thermometer sensor during the measurements, the uncertainty in the immersion depth of the thermometer due to the uncertainty in the exact fraction of the metal sample frozen, and the uncertainty in the adequacy of immersion of the thermometer to eliminate the thermometer stem conduction during the fixed-point cell comparisons. The uncertainty in the immersion depth due to the uncertainty in the position of the (HT)SPRT sensor was estimated to be 1 mm. This uncertainty is equivalent to a minimum of about 0,4 μK at the TPW and a maximum of about 3,2 μK at the freezing point of Ag. The uncertainty in the fraction of metal frozen was estimated to be about 5 % relative to the estimated 20 % fraction frozen during the comparisons. This leads to an uncertainty in the immersion depth of the thermometer that is equivalent to a minimum of about 0,2 μK at Ga and Sn and a maximum of about 1,6 μK at the freezing point of Ag. Since the thermometers had sufficient immersion to track the ITS-90 specified hydrostatic-head effect in the cells to within the scatter of the measurements (see Figure 2), the uncertainty in the adequacy of immersion of the thermometer to eliminate the thermometer stem conduction was estimated from the residuals of a least-squares fit of the immersion data to the predicted curve. These were calculated to be the equivalent of no more than 10 μK (at the Ag freezing point) and to as small as 3 μK (at the Ga melting point). The combination of these three sources of uncertainties gives a type B standard uncertainty at each fixed point as given in Table 4, which lists the type A and type B standard uncertainties, and the expanded uncertainties ($k=2$).

Table 4. Type A uncertainty (of the mean) u_A, type B standard uncertainty u_B and expanded uncertainty $U(k=2)$ associated with the NIST comparison measurements.

Fixed point		u_A/μK	u_B/μK	U/μK
Ag	FP	23	15	55
Al	FP	17	12	42
Zn	FP	13	13	37
Sn	FP	10	9	27
In	FP	13	12	35
Ga	TP	3	5	12
H_2O	TP	7	6	18

5. Results and discussion

The results of the comparisons of the fixed-point cells relative to the NIST reference cells are presented in Table 3. The results have been corrected for differences in the cell pressures and hydrostatic heads. The result of the comparison of NIST cell Sn 75A with the current Sn reference cell, Sn 88A, is listed there also. Also listed are the results of the chain of comparisons of NIST cell Zn 86D to the current Zn reference cell, Zn 93-4, as are the results of the chain of comparisons of NIST Al cell A-2 with the current Al reference cell, Al 94-2. Additionally, the results of the chain of comparisons of

NIST cell Ag 79-1 with the current NIST Ag reference cell, Ag 92-1, are listed.

For the TPW cells, the results presented in Table 3 were obtained from ratios of the extrapolated zero-power value of the bridge readings of the SPRT in the test cell to that of the extrapolated zero-power value determined from the first set of bridge readings of the SPRT in the NIST reference cell.

For the Ga, In, Sn, Zn and Al fixed points, the W values (where, for example, $W(\mathrm{Zn}) = R(\mathrm{Zn})/R(\mathrm{TPW})$ were calculated by using the resistance of the SPRT at the TPW [R(TPW)] obtained just after the particular comparison experiment. This ensured that the oxidation state of the Pt sensor of the SPRT was the same when measurements were made at the TPW as it was when measurements were made at the metal fixed point.

The W(Ag) values for silver, however, were calculated by using the R(TPW) obtained just prior to the particular comparison experiment. With these R(TPW) values, the spread of the W(Ag) values for the VNIIM-made HTSPRT in NIST cell Ag 92-1 in comparison experiments with cells CMS/ITRI CMS Ag 92-1 and PTB Ag 6 were $0{,}1_4$ mK and $0{,}1_1$ mK, respectively. Although, in general, an average of the two R(TPW) values might be a better choice, in this case we believe that the R(TPW) obtained just prior to the comparison experiment is better since the temperature of NIST cell Ag 92-1 was unchanged during the course of the comparisons, and, consequently, the first W(Ag) values obtained on the freezing curves for the NIST cell throughout the comparison experiments should have been constant. This feature was exemplified better when values of W(Ag) were calculated by using the R(TPW) value obtained just prior to a given comparison experiment instead of using the average of that R(TPW) value and that obtained just after the comparison.

The IMGC cells Al Co1 and Ag Lei3 compared at NIST with NIST cells were sealed transfer cells that were used for an indirect comparison of the NIST and IMGC Al and Ag *reference* cells. These transfer cells were compared at NIST directly with the NIST cells using the technique described in this paper. The same transfer cells were also compared (by IMGC) at IMGC with the IMGC reference cells, but not by means of a direct comparison as at NIST because IMGC did not have two identical furnaces available at that time.

Table 5. Observed temperature differences between IMGC fixed-point cells [2] expressed as $\Delta T = [T(\text{transfer}) - T(\text{reference})]$.

Laboratory	Fixed point	Cell serial number		ΔT/mK
		Reference	Transfer	
IMGC	Al FP	Al Co3	Al Co1	− 0,09
	Ag FP	Ag Co2	Ag Lei3	− 4,96

The technique used by IMGC involved making several measurements with several (HT)SPRTs on a given cell in a given furnace, and then comparing those results with similar ones obtained in the same furnace using the other cell. Consequently, the uncertainties in the measurements for the IMGC comparisons of their cells are somewhat larger than those quoted in Section 4 of this paper for the NIST measurements. The expanded uncertainties ($k = 2$) for the IMGC measurements were 0,12 mK and 0,75 mK at the Al and Ag fixed points, respectively. The results of the IMGC comparisons of their Al and Ag cells are given in Table 5.

After the comparisons of the NIST and IMGC cells, the same Al Co1 and Ag Lei3 transfer cells were circulated in Europe for an interlaboratory comparison [2].

Using the results presented in Tables 3 and 5, the differences of the temperatures of the NIST and IMGC reference cells are

$$T\ (\text{IMGC cell Al Co3}) - T\ (\text{NIST cell Al 94-2}) = -0{,}71\ \text{mK}$$

and

$$T\ (\text{IMGC cell Ag Co2}) - T\ (\text{NIST cell Ag 92-1}) = -3{,}85\ \text{mK}.$$

Also, from the results presented in Tables 3 and 5, the differences between the fixed-point temperatures of the cells of the other participants may be determined. These are presented in Table 6.

Direct comparison of fixed-point cells, as conducted at NIST and presented in this paper, is the best method for the determination of temperature differences between cells since it is capable of yielding the smallest uncertainties of all methods. It minimizes changes in the (HT)SPRT and in the measurement system that might

Table 6. Indirect results of fixed-point cell comparisons. The values for the differences in temperature of the cells are expressed as $\Delta T(\text{Lab 1, Lab 2}) = T(\text{Lab 1}) - T(\text{Lab 2})$.

	ΔT/mK									
Lab 1	CMS	IMGC	NIM	PTB	UME	CMS	PTB	UME	UME	CMS
Lab 2	CENAM	CENAM	CENAM	CENAM	CENAM	IMGC	IMGC	IMGC	NIM	PTB
H_2O TP			0,00		− 0,04				− 0,04	
Sn FP		− 0,07			− 0,63			− 0,56		
Zn FP		0,17								
Al FP		0,05								
Ag FP	− 2,74	− 3.60		0,03		0,86	3,63			− 2,77

occur if cells were to be assessed by the technique of comparing the freezing and/or melting curves of one cell with those of another (and of the $W(t)$ values obtained therefrom), with the measurements on the cells being made in the same furnace, but at separate times, for example. In direct comparisons of fixed-point cells, either W or resistance values of the (HT)SPRT may be used. The use of W values is preferable, however, since this provides a check on the stability of both the thermometer and the fixed-point cell.

It should be emphasized that although the results of the comparisons of the metal fixed-point cells reported here are, in general, excellent, that does not mean that realizations of the fixed-point temperatures with those cells will be highly accurate under all circumstances. For example, if the hydrostatic-head effect measured in the cell with the (HT)SPRT to be used in the realization of the fixed-point temperature is not in agreement with the predicted effect because there is an appreciable temperature gradient over the volume of the sample or because the fixed-point-cell assembly is inadequately constructed, then the realization almost certainly will be in error, and the error may be substantial.

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